Docket No.: 27617-00003-US
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Masahiko Matsukawa et al.

Application No.: 10/743,387 Confirmation No.: 7938

Filed: December 23, 2003 Art Unit: 1742

For: PRETREATMENT METHOD FOR COATING Examiner: L. L. Zheng

DECLARATION UNDER 37 CFR 1.132

MS Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

I, Masanobu Futshuhara, a citizen of Japan, declare as follows:

1) I am one of the inventors of the above-identified application and am familiar with the subject matter thereof and am employed in the Surface Treatment Division of the Nippon Paint Co., Ltd.;

2) The following experiments were conducted by me and/or under my direction and/or supervision.

3) The following experiments demonstrate that the treatment agent of the present invention can form a conversion coat on a metal base material, in contrast to the Solution S2 of Experiment 4 of JP S59-064781 which cannot form a conversion coat on a metal base material.

4) The following experiments also demonstrate that the conversion coat formed on the metal base material using the treatment agent of the present invention is excellent

in adhesiveness with a cationic electrodeposition coat, and therefore, that the corrosion resistance of the metal base material is improved.

5) Methods and Results of the Experiments

5.1) Manufacture of the Test Plate

5.1.1 Manufacture of the Test Plate of the Present Invention

An Admixture was formed by adding 5 parts by mass of water (ion exchange water) to 95 parts by mass or γ-aminopropyltriethoxysilane (KBM903 manufactured by Shin-Etsu Chemical Co., Ltd.; hereinafter referred to as "APS") followed by stirring, and then allowing to stand until transparent (step b1).

0.2 g of the admixture and 1.1 g of fluorozirconic acid (H_2ZrF_6) were added to 1000g of water to prepare solution S1 (pH 4.2) (step b2).

Degreased cold rolled steel sheet for automobile use (manufactured by Paltec Test Panels Co., Ltd.) was dipped into the solution S1 at 40°C for 120 seconds (step b3).

Thereafter, the steel sheet was immediately washed with water without drying (step c), followed by cationic electrodeposition coating (PN110G manufactured by Nippon Paint Co., Ltd; 25 μm of film thickness) (step d) and heating at 170°C for 20 minutes to obtain a test plate.

5.1.2 Replicate of Example 4 of JP S59-064781A

According to Example 4 of JP S59-064781A, 10 g of APS (KBM903), and 1 g of zircon hydrofluoric acid were mixed, and then water was added thereto to adjust the volume to 1 L, to prepare the solution S2 (pH 10.0) without conducting the step b1 of the "Manufacturing of the Test Plate of the Present Invention".

The cold rolled steel sheet for automobile use using the solution S2 as described above, was then washed with water without drying, followed by cationic electrodeposition coating and heating.

5.2 Evaluating the Amount of Coating Film, and Discussion

The amount of coating film formed on the surface of each of the steel sheets was measured by fluorescent X ray spectrometry, and the composition of each of the coating films was analyzed. The results are shown in Table 1.

As shown in Table 1, zirconium was not detected on the surface of steel sheet when the solution S2 of Example 4 described in JP S59-064781A was used for surface treatment.

In contrast, when the solution S1 of the present invention was used for surface treatment, zirconium was detected, thus showing that zirconium is included in the coating film, and in addition, the silicon was also detected.

Therefore, as zirconium is washed away in the water washing step when the solution S2 of Example 4 of JP S59-064781A was used, the conversion coating is thought not to be formed on the surface of the steel sheet.

In contrast, as zirconium is not washed away even in the water washing step when the solution S1 of the present invention was used, the conversion coating containing zirconium was thought to be formed on the surface of the steel sheet. Moreover, as silicon was also detected in the coating film, the conversion coating film is thought to contain APS.

5.3 Evaluating the Adhesiveness of the Coating Film (SDT), and Discussion

The corrosion resistance of the obtained electrodeposition coating was evaluated by a warmed salt solution dipping test. In other words, a single lesion 10 cm long was incised in the electrodeposition coating by a cutter such that the lesion reached the metal base material, and the metal material was dipped into a 5 wt. % NaCl solution at 55 °C for 240 hrs. After the dipping, tape detachment test was conducted using the adhesive tape (CT405AP-12, manufactured by Nichiban Co., Ltd, 12 mm width), the detachment width of the coating film from the site of the lesion was measured. The results are shown in Table 1.

As shown in Table 1, the SDT detachment width of the coating film formed on the steel sheet treated with the solution S2 of Example 4 of the JP S59-064781A was determined to be 12 mm (the same width as the adhesive tape), indicating complete detachment.

In contrast, the SDT detachment width of the coating film formed on the steel sheet treated with the solution S1 of the present invention was determined to be 1 mm, indicating almost no coating film detachment.

Therefore, the adhesiveness between the electrodeposition coating film of the present invention and the steel sheets is thought to be extremely excellent, and the corrosion resistance of the steel sheet is thought to improve accordingly.

[Table 1]

Treatment Solution	Composition of the Treatment Solution/ Treatment Condition						Composition of the Coating film		SDT detachment width (mm)
	H_2ZrF_6 (g/L)	APS (g/L)	Hydrolyzed APS Solution (g/L)	pH	Temp. (°C)	Time (sec.)	Zr (gm/m^2)	Si (gm/m^2)	
S1 (Present Invention)	1.1	-	0.2	4	40	120	35.1	3.1	1
S2 (Example 4 of the JP S59-064781A)	1	10	-	10	40	120	0.1	0.4	12 mm (same with the width of detachment tape)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: 29th Oct. 2009

By: Masanobu Futsuhara

Masanobu Futsuhara